（※本報告書は英語で記述してください。ただし、産業利用課題として採択されている方は日本語で記述していただいても結構です。）

# MLF Experimental Report

| | |
|---|---|
| 課題番号 Project No.<br>2017A0032 | 提出日 Date of Report |
| 実験課題名 Title of experiment<br>Measurement of anisotropy of residual stress and strain in Ni-based superalloys due to precipitation misfit and plastic strain | 装置責任者 Name of responsible person<br>Stefanus Harjo |
| 実験責任者名 Name of principal investigator<br>Tokujiro Yamamoto | 装置名 Name of Instrument/（BL No.）<br>TAKUMI /（BL19） |
| 所属 Affiliation<br>Utsunomiya University | 実施日 Date of Experiment<br>April 2 – April 5 |

試料、実験方法、利用の結果得られた主なデータ、考察、結論等を、記述して下さい。（適宜、図表添付のこと）

Please report your samples, experimental method and results, discussion and conclusions. Please add figures and tables for better explanation.

## 1. 試料 Name of sample(s) and chemical formula, or compositions including physical form.

Change in the diffraction peaks of nickel-based superalloy (PWA1480) specimens was examined with applying tensile stress at high temperature. The specimens were composed of the FCC γ phase and $L1_2$ γ' phase. The γ' phase was precipitated in the γ phase with keeping crystallographic orientation between the γ phase by aging at high temperature. The morphology of the γ matrix and the γ' precipitates in the specimens is schematically illustrated in Fig. 1. When the longitudinal direction of a round bar single crystal specimen before aging is $[001]_\gamma$, three types of the thin plate-like γ channels are formed between the γ' precipitates. Here, the γ channel parallel to $(001)_\gamma$ (also parallel to $(001)_{\gamma'}$) is denoted as the (001) γ channel, for example.

The lattice parameters of the γ and γ' phases are $a_0$ and $a$, where $a$ is slightly smaller than $a_0$. Because of the misfit between the γ matrix and the γ' precipitates, residual stress exists in the specimen. According to analysis using micromechanics, tensile stress is applied to the thickness direction and compression stress is applied perpendicular to the thickness direction by precipitation of the γ' phase.

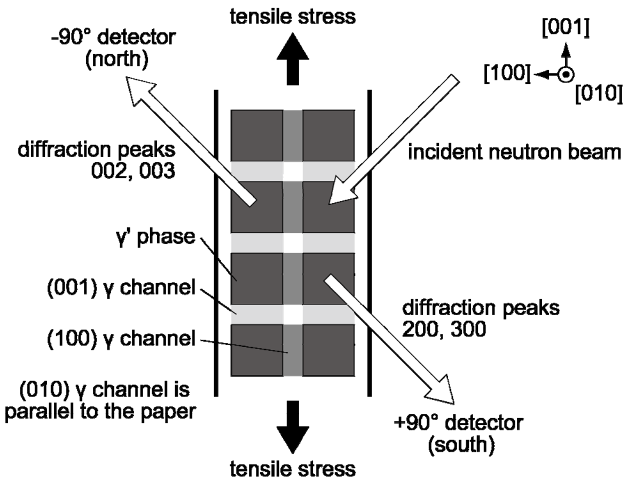


Fig. 1. Schematic illustration of the experimental setup for neutron diffraction experiments.

2. 実験方法及び結果（実験がうまくいかなかった場合、その理由を記述してください。）
Experimental method and results.　If you failed to conduct experiment as planned, please describe reasons.

The round bar single crystal specimens were mounted to a tensile testing machine installed in BL19 Takumi beamline, so that tensile stress can be applied along the longitudinal direction, i.e. $[0\ 0\ 1]_{\gamma}$. The incident neutron beam was radiated along $[1\ 0\ -1]_{\gamma}$. The specimens were rotated to detect 0 0 *l* and *h* 0 0 diffraction peaks (see Fig. 1). Neutron diffraction patterns were measured using a time-of-flight method at room temperature, 673 K and 973 K. Measurements were also performed at 973 K with applying tensile stress to the specimens. The maximum tensile strain was 8%. Diffraction peaks of a pure Ni single crystal was also measured without applying stress at room temperature as a reference. 0 0 2 and 2 0 0 peaks composed of the γ and γ' phases, while 0 0 3 and 3 0 0 diffraction peaks came only from the γ' phase.

Fig. 2 shows the profiles of the 0 0 3 and 3 0 0 diffraction peaks as a function of the specimen temperature. Both the lattice spacing of $(0\ 0\ 3)_{\gamma'}$ and $(3\ 0\ 0)_{\gamma'}$ increased with increasing the temperature because of thermal expansion of the specimen.

After heating up to 973 K, tensile stress was applied to the specimen along $[0\ 0\ 1]_{\gamma}$ orientation, and followed by unloading. Fig. 3 shows the profiles of the 0 0 3 and 3 0 0 diffraction peaks at 973 K after unloading as a function of the plastic strain introduced in the specimen. The lattice spacing of the $(0\ 0\ 3)_{\gamma'}$ increased with increasing the plastic strain, while that of the $(3\ 0\ 0)_{\gamma'}$ decreased. It represents that tensile and compression stress was applied along [0 0 1] and [1 0 0] orientation of the γ' precipitates, respectively.

It should be noted that the width of the 0 0 3 diffraction peak increased more rather than the peak position upon tensile testing. Meanwhile, the width of the 3 0 0 diffraction peaks also increased. These results indicate that a certain amount of plastic strain was introduced in the γ' precipitates.

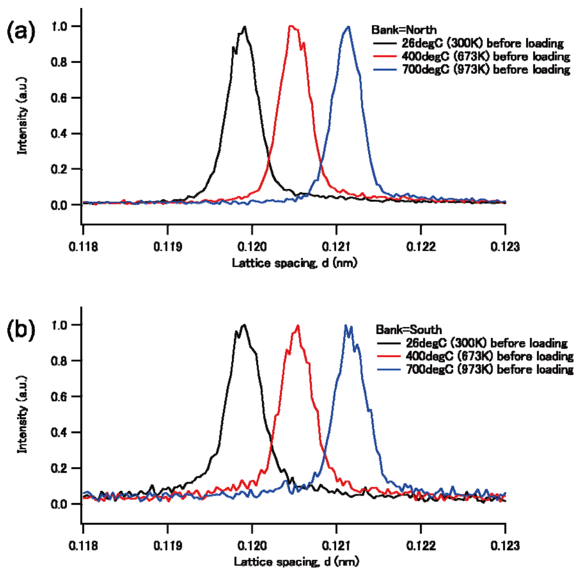


Fig. 2. Change in the lattice spacing of (a) $(003)_{\gamma'}$ and $(300)_{\gamma'}$ plane before loading as a function of the specimen temperature.

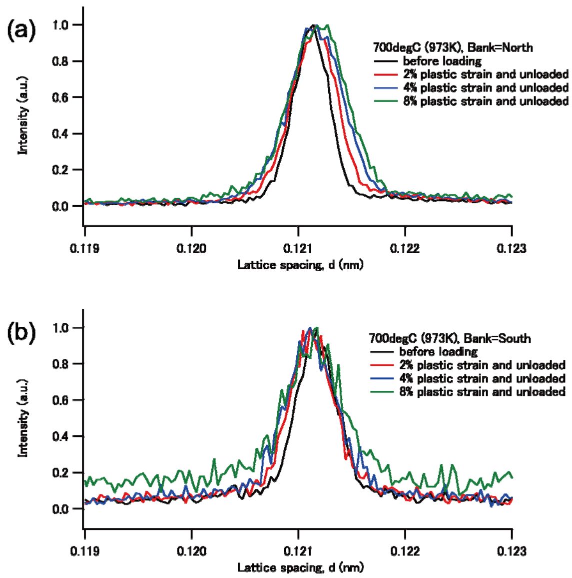


Fig. 3. Change in (a) 003 and (b) 300 diffraction peaks of the γ' phase at 973 K after unloading as a function of the plastic strain introduced in the specimen.

## 2. 実験方法及び結果（つづき） Experimental method and results (continued)

For the 0 0 2 and 2 0 0 diffraction peaks, the lattice spacing of the (0 0 2) and (2 0 0) planes of the $\gamma$ matrix and $\gamma'$ precipitates also increased by thermal expansion, as shown in Fig. 4.

However, even though plastic strain was introduced in the specimen, the change in the position of the 0 0 2 and 2 0 0 diffraction peaks, which is composed by both the $\gamma$ matrix and $\gamma'$ precipitates, was smaller than that of the 0 0 3 and 3 0 0 peaks of the $\gamma'$ precipitates as shown in Fig. 5.

Bruno et al. (2003) analyzed the 0 0 2 and 2 0 0 diffraction peaks, assuming that those peaks were composed of three peaks. However, according to our analysis using micromechanics, the $\gamma$ matrix is also affected by the residual stress caused by the misfit. For example, the 0 0 2 diffraction peak should be composed by the peaks caused by the following four lattice planes:

$(0\ 0\ 2)_{\gamma}$ with tensile stress in the (0 0 1) $\gamma$ channel,

$(0\ 0\ 2)_{\gamma}$ with compression stress in the (1 0 0) $\gamma$ channel,

$(0\ 0\ 2)_{\gamma}$ with compression stress in the (0 1 0) $\gamma$ channel,

$(0\ 0\ 2)_{\gamma'}$ with tensile stress in the $\gamma'$ precipitates

Therefore, the 0 0 2 diffraction peaks is decomposed to the four peaks by the $(0\ 0\ 2)_{\gamma}$ and $(0\ 0\ 2)_{\gamma'}$ planes, and the 2 0 0 diffraction peaks is also decomposed to the four peaks by the $(2\ 0\ 0)_{\gamma}$ and $(2\ 0\ 0)_{\gamma'}$ planes. The asymmetry and tails of the 0 0 2 and 2 0 0 diffraction peaks is agree with the decomposed four peaks above mentioned.

Our micromechanics analysis revealed that the change in the lattice spacing for $(0\ 0\ 2)_{\gamma}$ with tensile and compression stress is about 3%. The composition of the four diffraction peaks in the asymmetrical 0 0 2 and 2 0 0 diffraction peaks will be examined, taking the change in the lattice spacing into consideration.

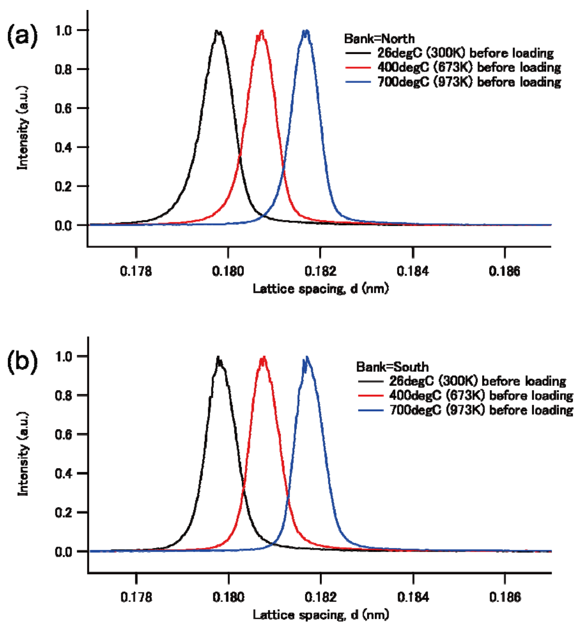


Fig. 4. Change in the lattice spacing of (a) 002 and 200 diffraction peaks before loading as a function of the specimen temperature.

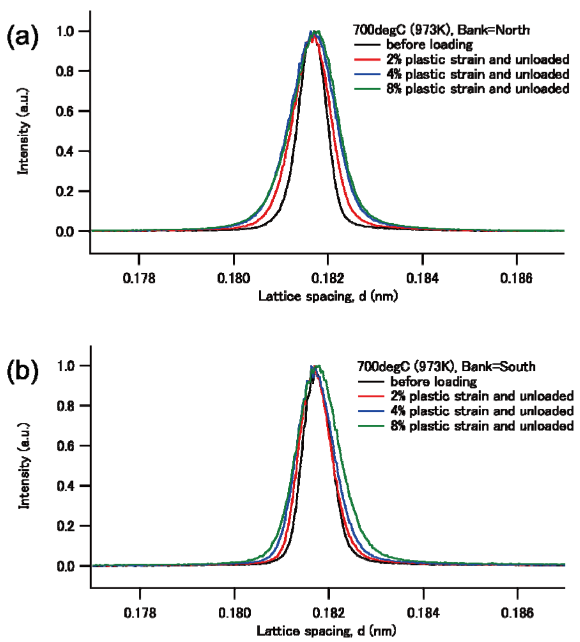


Fig. 5. Change in (a) 002 and (b) 200 diffraction peaks at 973 K after unloading as a function of the plastic strain introduced in the specimen.